

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau



(43) International Publication Date
26 June 2003 (26.06.2003)

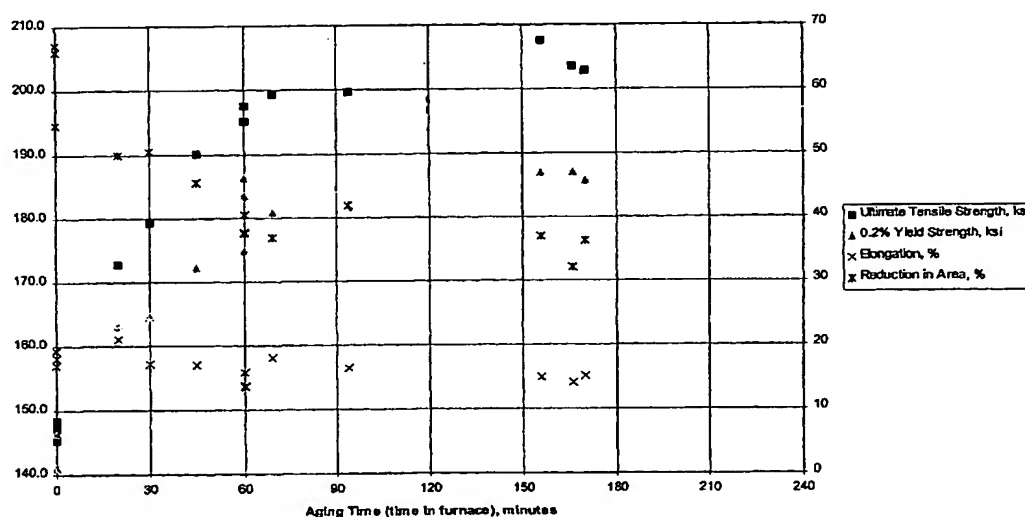
PCT

(10) International Publication Number
WO 03/052155 A1

- (51) International Patent Classification⁷: C22F 1/18 (74) Agents: VICCARO, Patrick, J.; Allegheny Technology Incorporated, 1000 Six PPG Place, Pittsburgh, PA 15222, et al. (US).
- (21) International Application Number: PCT/US02/18269
- (22) International Filing Date: 7 June 2002 (07.06.2002) (81) Designated States (*national*): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZM, ZW.
- (25) Filing Language: English
- (26) Publication Language: English
- (30) Priority Data:
60/340,671 14 December 2001 (14.12.2001) US
10/165,348 7 June 2002 (07.06.2002) US
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- (84) Designated States (*regional*): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).
- Published:
— with international search report

[Continued on next page]

(54) Title: METHOD FOR PROCESSING BETA TITANIUM ALLOYS



(57) Abstract: An embodiment of the present invention comprises processing a beta titanium alloy by a method including the steps of cold working the alloy and then direct aging the alloy for a total aging time of less than 4 hours. The method may include fabricating the alloy into the article of manufacture such as, for example, a bar, wire, or a coil spring. The method may be utilized to produce articles with high tensile strength while retain ductility. The beta titanium alloy may be any beta titanium alloy, for example, the alloy comprising, by weight, 3.0 % to 4.0 % aluminium, 7.5 to 8.5 % vanadium, 5.5 to 6.5 % chromium, 3.5 to 4.5 % molybdenum, 3.5 to 4.5 % zirconium, and titanium. The alloy may be hot worked, cold worked to provide a 5 to 60 % reduction, and irect aged for a total time of less than 4 hours.



WO 03/052155 A1



For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

METHOD FOR PROCESSING BETA TITANIUM ALLOYS

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FIELD OF INVENTION

The invention relates to a method for processing titanium alloys and, more particularly, beta titanium alloys. The method of the present invention includes cold working a beta titanium alloy and subsequently direct aging the alloy for less than 4 hours.

DESCRIPTION

The unique properties of titanium alloys allow their use in applications requiring high corrosion resistance, high strength, and low material weight. For cost reasons, applications requiring corrosion resistance often utilize low-strength unalloyed titanium mill products. Unalloyed titanium may be fabricated into equipment used in, for example, chemical processing, desalination, and power generation. In contrast, high performance applications often utilize high-strength titanium alloys in a very selective manner depending on several design factors including weight, strength, ductility, and reliability requirements. To meet the requirements of their specialized uses, alloys intended for high performance applications normally are more stringently processed, with resulting additional cost, than titanium for corrosion service. Nevertheless, the combination of high strength and stiffness, favorable toughness, low density, and good corrosion resistance inherent in various titanium alloys useful in low to moderate temperature applications allows substantial weight savings in aerospace structures and other high-performance applications. Such weight savings often justify the increased costs associated with processing the titanium alloys.

A titanium alloy may be classified as one of several metallurgical types, for example, alpha, near-alpha, alpha-beta, or beta. Beta titanium alloys are particularly useful in aerospace structures. Hot worked beta titanium alloys may be cold worked to final or near final form. The cold working process imparts high strength levels and/or a favorable ductility/strength relationship in the alloys. Certain "Aerospace Material Specifications", AMS 4957A and AMS 4958A, define recommended processing conditions for the beta titanium alloy Ti-3Al-8V-6Cr-4Zr-4Mo (referred to herein as Ti-38-644 alloy) to produce round bar or wire primarily for use as aerospace coil springs. Typically, aerospace spring applications require high tensile strength, low density and corrosion resistance. Ti-38-644 alloy includes, by weight, 3.0 to 4.0% aluminum, 7.5 to 8.5% vanadium, 5.5 to 6.5% chromium, 3.5 to 4.5% molybdenum, 3.5 to 4.5% zirconium, maximum 0.14% oxygen, maximum 0.05% carbon, maximum 0.03% nitrogen, and remainder titanium. AMS 4957B requires certain additional restrictions on alloy composition, including maximum 0.30% iron, maximum 0.10% palladium, maximum 300 ppm hydrogen, maximum 50 ppm yttrium, and maximum total residual elements 0.40%. According to the AMS specifications, the alloy is aged by heating to a temperature within the range of 850°F to 1050°F (454°C to 566°C) and held at the selected temperature $\pm 10^\circ\text{F}$ (6°C) for six to twenty hours. The required minimum tensile properties, determined according to ASTM E8 or ASTM E8M, as applicable, depend on the nominal diameter of the round bar or wire final product, but in no case are to be less than minimum tensile strength of 180 ksi, minimum elongation of 8%, and minimum reduction of area ("RA") of 20%.

Whether a titanium alloy is of the alpha, near-alpha, alpha beta, or beta metallurgical type is influenced by the chemical composition of the alloy, the applied heat treatment, and other factors. The metallurgical type designations refer to the predominant crystalline phase present in the microstructure of the alloy at room temperature. Titanium metal has a close packed hexagonal crystal structure ("hcp"), referred to as "alpha", at room temperature. This structure may be transformed to a body-centered cubic ("bcc") crystal structure ("beta") at elevated temperatures. The temperature at which this transformation occurs is referred to as the "beta transus temperature". The beta transus temperature for a

commercially pure titanium alloy is approximately 1625°F (885°C). Certain alloying elements added to pure titanium promote the formation of one or the other of the alpha and beta crystal structures. Elements that favor the alpha structure are referred to as "alpha stabilizers", and elements that favor the beta structure are referred to as "beta stabilizers". Aluminum, for example, is an alpha stabilizer and, therefore, adding aluminum to a titanium alloy increases the beta transus temperature. Chromium, iron, molybdenum, and vanadium are beta stabilizers, and their addition lowers the beta transus temperature, stabilizing the beta structure at lower temperatures. The relative amounts of alpha and beta stabilizers in an alloy and the heat treatment applied to the alloy determine whether the microstructure of the alloy is predominantly single alpha phase, single beta phase, or a mixture of alpha and beta phases over a particular temperature range.

The properties of a titanium alloy are related to its microstructure. Two-phase alpha-beta alloys generally exhibit tensile strengths greater than single-phase alpha alloys or single-phase beta alloys. Also, alpha-beta alloys can be further strengthened by heat treatment because the microstructure may be manipulated by controlling heating, quenching, and aging cycles.

Many beta titanium alloys are alloyed with more than one beta stabilizer. With sufficient quantities of beta stabilizer, and suitable control over heat treatment and cooling, beta phase may be retained at relatively low temperatures, below the alloy's normal beta transus temperature. For example, beta phase may be retained in a titanium alloy by rapid cooling from above and through the beta transus temperatures, such as by quenching. However, the titanium alloy must have sufficient quantities of beta stabilizers to prevent the beta phase from transforming to alpha phase by martensitic transformation. Titanium alloys containing beta stabilizers in quantities sufficient to reduce the alloy's martensitic transformation temperature to below room temperature but not sufficient to reduce the beta transus to below room temperature are known as "metastable" beta titanium alloys. Metastable beta titanium alloys may maintain at least a portion of beta structure after heat treatment and cooling to room

temperature. As used herein, references to a beta titanium alloy are to a metastable beta titanium alloy as described above.

In addition, unless otherwise indicated, all numbers expressing quantities of ingredients, time, temperatures, and so forth used in the present specification and claims are to be understood as being modified in all instances by the term "about." Accordingly, unless indicated to the contrary, the numerical parameters set forth in the following specification and claims are approximations that may vary depending upon the desired properties sought to be obtained by the present invention. At the very least, and not as an attempt to limit the application of the doctrine of equivalents to the scope of the claims, each numerical parameter should at least be construed in light of the number of reported significant digits and by applying ordinary rounding techniques.

Notwithstanding that the numerical ranges and parameters setting forth the broad scope of the invention are approximations, the numerical values set forth in the specific examples are reported as precisely as possible. Any numerical value, however, may inherently contain certain errors necessarily resulting from the standard deviation found in their respective testing measurements.

An embodiment of the present invention comprises processing a beta titanium alloy by a method including the steps of cold working the alloy and then direct aging the alloy for a total aging time of less than 4 hours. The beta titanium alloy may be, for example, Ti-38-644 alloy. The method may include fabricating the alloy into an article of manufacture such as, for example, a bar, wire, a coil spring.

Another embodiment of the present invention is a method for producing a spring or other article of manufacture from a beta titanium alloy. The beta titanium alloy may be, for example, the alloy comprising, by weight, 3.0% to 4.0% aluminum, 7.5 to 8.5% vanadium, 5.5 to 6.5% chromium, 3.5 to 4.5% molybdenum, 3.5 to 4.5% zirconium, and titanium. The alloy is hot worked, cold worked to provide a 5% to 60% reduction, and direct aged for a total time of less than 4 hours. As used herein, cold working is defined as various working processes performed at a temperature below an effective aging temperature of

the alloy. Cold working of a titanium alloy, therefore, may be performed at temperatures below the beta transus temperature of the alloy. Cold working permanently deforms the work piece, which does not return to its original shape when the load causing the deformation is removed. The degree of cold working, typically, is determined by the percentage reduction in cross-sectional area of the work piece. Thus, a 5% reduction achieved by cold working refers to a reduction of 5% in the cross-sectional area of the work piece upon cold working. Any cold working technique may be used in embodiments of the present invention. Useful cold working techniques include, but are not limited to, compression processes, drawing, wire drawing, tube drawing, deep drawing, rolling, contour forming, extruding, cold heading, swaging, coining, forging, tension processes, stretch forming, and spinning.

Cold working may be used to improve the mechanical properties of an alloy including hardness, yield strength, and tensile strength. Ductility, however, may be reduced during cold working. Ductility is a measure of the ability of a material to deform plastically without fracture. Elongation or RA in tensile testing typically is used as a measure of ductility of material. The method of the present invention may be used to increase the strength of beta titanium alloys while also maintaining good ductility and significantly increasing the aging response of the alloy.

A beta titanium alloy was prepared and processed according to the method of the present invention. Its properties were then compared with the same alloy composition processed using a conventional method including cold working and heat treating steps. This testing is described in greater detail below:

A melt of a Ti 38-644 alloy was prepared and cast into an ingot. The alloy had the average composition, in weight percentage, shown in Table 1. A first ingot was hot rolled at a temperature not to exceed 1750°F, annealed and air cooled.

Ti	Al	V	Cr	Zr	Mo	O	Fe	C	N
Bal.	3.42	7.84	5.95	3.98	4.15	0.08	0.13	0.01	0.006

Table 1: Composition of first Ingot

A portion of the hot rolled, annealed, and air cooled ingot was processed by the method of the present invention. Another portion of the hot rolled, annealed, and air cooled ingot was processed in a conventional manner for comparison purposes. The portion processed in the conventional manner was hot worked, then solution heat treated, and subsequently aged. The heat treatment parameters were varied to assess the impact on mechanical properties. As is known in the art, solution heat treating is a heat treatment step wherein an alloy is heated to a suitable temperature and held at the temperature for a time period sufficient to cause one or more constituents of the alloy to enter solid solution. The alloy is then cooled rapidly so as to hold the one or more constituents in solution. Solution heat treatment typically is performed on an alloy to improve ductility at a given strength.

Several variations of the conventional heat treatment process were compared with the process of the present invention. Table 2 includes the results of room temperature tensile testing of the alloy of Table 1 processed by the conventional heat treatment process under various conditions. All tensile properties reported in Table 2 were determined in accordance with ASTM E 8. Tensile testing was used to determine ultimate tensile strength ("UTS"), 0.2% yield strength, elongation, and RA of the test pieces. RA and elongation are measures of ductility of the test pieces. Elongation is the amount of extension of a test piece when stressed. In tensile testing, elongation is the increase in gage length, measured after fracture of the specimen with the gage length, usually expressed as a percentage of the original gage length as marked on the test piece.

Room Temperature Tensile Data As-Rolled and Heat Treated

Solution Heat Treatment Temp. & Time at temperature	Cooling After Solution	Aging Temp. & Time	Cooling After Age	UTS (ksi)	0.2% YS (ksi)	Elong. (%)	RA (%)	Modulus
as-rolled	as-rolled	as-rolled	As-rolled	128	126	30	67	12.5
1400°F / 1 hr	water quench	None	None	130	128	24	61	12.8
1400°F / 1 hr	water quench	900°F / 8 hr	air cool	148	138	17	47	13.8
1400°F / 20 min	air cool	900°F / 8 hr	air cool	174	160	17	37	14.1
1400°F / 20 min	air cool	900°F / 16 hr	air cool	193*	180	5*	3*	14.9
1400°F / 20 min	air cool	900°F / 24 hr	air cool	193*	179	5*	4*	14.8
1400°F / 20 min	air cool	950°F / 8 hr	air cool	167	155	20	46	13.8
1400°F / 20 min	air cool	950°F / 16 hr	air cool	184	170	18	43	14.9
1400°F / 20 min	air cool	950°F / 24 hr	air cool	186	174	14	35	14.8

* = failed near punch mark

Table 2: Properties of Conventionally Processed Ti-38-644 Alloy

The test pieces listed in Table 2 were hot rolled from 4 inch diameter billet to 0.569 inch diameter bar and solution heat treated prior to aging. The data in Table 2 clearly shows that long aging times, more than 8 hours, are required to achieve high strength, greater than 180 ksi, in the alloy. For both of the tested solution heat treatment processes (1400°F (760°C) for one hour and 1400°F (760°C) for 20 minutes), the conventional process required more than 8 hours of aging to achieve the minimum tensile strength for Ti-38-644 bar and wire specified in AMS 4957A and AMS 4958B. AMS 4958A specifies that the beta titanium alloy must receive no more than 5% cold work reduction after hot rolling and solution heat treating. AMS 4958A also requires that the alloy be subjected to aging temperatures for at least 12 hours. Additionally, due to solution heat treatment and aging at elevated temperatures, an oxide layer may form on the alloy surface. AMS 4958A requires an acid pickling step to remove this layer.

DESCRIPTION OF EMBODIMENTS OF THE INVENTION

The aging time of an alloy may be measured and expressed by different criteria. For example, the length of the aging process may be measured as the total time the alloy is exposed to the aging temperature in a furnace, or as the total time that the surface or an internal portion of the alloy is maintained within the aging temperature range. Unless otherwise noted, all aging times

reported herein for embodiments of the present invention are the total time that the alloy is exposed to an environment at approximately the desired aging temperature. Aging of the test piece samples listed in the examples was performed in a laboratory oven. More efficient means for heating an alloy, such as, for example, a production convection oven, may allow faster heat transfer to the alloy and thereby reduce the minimum aging time necessary to impart desired properties to the alloy. The method of the present invention is not limited to the embodiments described herein, including the particular aging equipment used, but includes various other embodiments. Thus, the embodiments of the present invention presented herein are only examples of the invention, and the scope of the invention is not so restricted.

An embodiment of the process of the present invention includes direct aging a beta titanium alloy for less than 4 hours after a step of cold working. Prior to cold working, the beta titanium alloy may be hot worked. After hot working, and prior to cold working, the alloy also may be annealed. A preferred annealing temperature for beta titanium alloys is 1425°F (774°C). Strength and ductility has been shown to be nearly identical for test pieces that have been annealed and test pieces that have not been annealed prior to cold working and aging by the process of the present invention.

The features and advantages of embodiments of the present invention may be better understood by reference to the accompanying figures, in which:

Figure 1 is a graph depicting the effect of aging time on UTS, 0.2% yield strength, elongation, and RA of a Ti-38-644 alloy subject to 13% or 15% cold work reduction and aged at 950°F (510°C);

Figure 2 is a graph depicting the effect of aging time and aging temperature on UTS of a Ti-38-644 alloy subjected to 13% or 15% cold work reduction and aged at 950°F (510°C), 1000°F (538°C) and 1050°F (566°C); and

Figure 3 is a graph depicting the effect of aging time and aging temperature on the RA of a Ti-38-644 alloy subjected to 13% or 15% cold work reduction and aged at 950°F (510°C), 1000°F (538°C), or 1050°F (566°C).

Test pieces of the alloy of Table 1 were processed according to the method of present invention. It will be understood that the method, of the present invention is applicable to other alloy compositions and is not limited to the application of the method described herein. By employing the present invention, a relatively high strength beta titanium alloy may be produced in a relatively short time while maintaining ductility. Embodiments of the present invention are listed in Tables 3-9. In each case, the test pieces were direct aged for a total aging time of less than 4 hours after a cold work step. Direct aging an alloy includes aging the alloy after working without an intermediate heat treating step, such as solution heat treating. Direct aging does not preclude other processing steps from being performed after cold working the alloy and prior to aging the alloy. These processes may be, for example, mechanical processes, such as shaving, or chemical processes, such as, pickling. The tables list the process steps employed and the mechanical properties of the processed alloy samples derived from tensile testing at room temperature.

Tables 3-9 list embodiments of the method of the present invention applied to the beta titanium alloy having the composition of Table 1. The amount of cold work may be to any degree and, preferably, in an embodiment of the method of the present invention the beta-titanium alloy is cold worked from at least a 5% reduction to a 60% reduction. Even more preferably, cold working the beta-titanium alloy comprises less than a 35% reduction. More preferably, an embodiment of the method of the present invention includes cold working the beta-titanium alloy to a reduction between 15% and 35%. With regard to Table 3, the test pieces were hot rolled, cold drawn to provide 8% reduction and then direct aged at the temperatures and for the times shown in the tables. The test pieces described in Table 3 also were annealed and centerless ground prior to cold drawing. The embodiments listed in Table 3 produced high strength (UTS greater than 170 ksi) and maintained ductility (greater than 8% elongation and greater than 20% RA) with less than four hours of direct aging. UTS values greater than 180 ksi and as high as 199 ksi were realized in the listed embodiments. The highest UTS values were realized at aging temperatures of 950°F (510°C), at which a UTS of 199 ksi was achieved at a total aging time of only 166 minutes.

The highest ductility as measured by elongation and RA was realized at the higher aging temperature of 1050°F (566°C).

Aging Temp. (°F) [°C]	Aging Time (minutes)	UTS (ksi)	UTS (MPa)	0.2% YS (ksi)	0.2% YS (MPa)	Elong. (%)	RA (%)	Modulus
-	0	140.5	969	132.5	913	19	61	12.0
950 [510]	166	199.0	1372	182.2	1256	14	41	14.4
950 [510]	170	197.5	1362	180.6	1245	14	35	13.4
1000 [538]	125	186.7	1287	168.7	1163	18	42	14.2
1000 [538]	200	186.0	1282	167.5	1155	18	41	14.9
1050 [565]	133	175.1	1207	156.9	1082	20	49	14.4
1050 [565]	182	172.8	1191	155.3	1071	21	52	14.5

Table 3: Tensile Testing Results for Embodiments of the Present Invention with 8% Cold Work Reduction

Table 4 lists embodiments of the present invention wherein test pieces were hot rolled, cold drawn to 13% reduction, and direct aged. Additionally, the embodiments described in Table 4 were annealed and centerless ground after hot rolling and prior to cold drawing. The embodiments of the method of the present invention in Table 4 displayed significantly increased strength after only 20 minutes of total aging time. With further aging at aging temperatures of 950°F (510°C) and 1000°F (538°C) the strength increased to a value greater than required by AMS 4958A and 4957B specifications. The test pieces aged at 1050°F (565°C), however, did not obtain strength as high as the test pieces aged at lower aging temperatures. The test pieces aged at 1050°F (565°C) did maintain a greater degree of ductility as measured by elongation and RA.

Aging Temp. (°F) [°C]	Aging Time (minutes)	UTS (ksi)	UTS (Mpa)	0.2% YS (ksi)	0.2% YS (MPa)	Elong. (%)	RA (%)	Modulus
as-drawn	As-drawn	145.3	1002	137.5	948	17	55	11.0
950 [510]	20	172.8	1191	163.1	1124	21	50	13.7
950 [510]	166	203.5	1403	187.1	1290	14	32	15.0
950 [510]	170	202.9	1399	185.8	1281	15	36	15.1
1000 [538]	20	168.7	1163	156.8	1081	24	51	14.4
1000 [538]	125	189.9	1309	172.1	1186	18	44	14.7
1000 [538]	200	189.8	1308	173.3	1195	16	41	15.0
1050 [565]	20	164.4	1133	151.3	1043	26	51	14.4
1050 [565]	133	178.7	1232	161.7	1115	20	47	14.4
1050 [565]	182	176.6	1217	159.3	1098	20	52	14.0

Table 4: Tensile Testing Results for Embodiments of the Present Invention with 13% Cold Work Reduction

Table 5 lists embodiments of the present invention wherein test pieces were hot rolled, cold drawn to 13% reduction, and direct aged, in a fashion similar to the embodiments shown in Table 4. However, the test pieces listed in Table 5 were not annealed and centerless ground prior to cold drawing. Nevertheless, the embodiments of the invention listed in Table 5 produced test pieces that exhibited high strength and ductility. The embodiments of Table 5 produced very high strength (UTS above 190 ksi) in the beta titanium alloy when aged for as short as 69 to 72 minutes. The results show that the annealing step may be excluded in embodiments of the present invention without significant affect on mechanical properties when the invention is applied to the beta titanium alloy of Table 1.

Aging Temp. (°F) [°C]	Aging Time (minutes)	UTS (ksi)	UTS (Mpa)	0.2% YS (ksi)	0.2% YS (MPa)	Elong. (%)	RA (%)	Modulus
as-drawn	As-drawn	147.2	1015	141.0	972	18	67	12.4
950 [510]	69	199.3	1374	181.0	1248	18	37	14.6
950 [510]	94	199.7	1377	181.7	1253	17	42	15.1
1000 [538]	72	194.7	1342	176.8	1219	20	43	14.5
1000 [538]	89	190.2	1311	173.3	1195	20	37	14.6
1000 [538]	125	190.8	1315	172.8	1191	16	45	14.5
1000 [538]	200	191.8	1322	173.8	1198	16	46	15.1
1050 [565]	81	179.0	1234	162.2	1118	24	57	15.0
1050 [565]	88	178.9	1233	161.6	1114	24	57	14.6

Table 5: Tensile Testing Results for Embodiments of the Present Invention with 13% Cold Work Reduction and Without Anneal

Table 6 lists embodiments of the present invention wherein test pieces were hot rolled, cold drawn to 15% reduction, and direct aged. Additionally, the test pieces of Table 6 were not annealed and centerless ground prior to cold drawing. Certain embodiments of the present invention in Table 6 included aging times less than 60 minutes. The embodiments including cold working to 15% reduction showed higher strengths than the embodiments including cold working to only 8% reduction, without a corresponding loss of ductility. The embodiments cold worked to 15% reduction achieved UTS greater than 190 ksi after aging for only 45 minutes of total aging time at 900°F (482°C) and 950°F (510°C), and achieved UTS greater than 200 ksi after aging for only 60 minutes of total aging time at the same temperatures.

Aging Temp (°F) [°C]	Aging Time (minutes)	UTS (ksi)	0.2% YS (ksi)	Elong. (%)	RA (%)
-	0	148.4	146.3	19.3	65.9
900 [482]	45	192.5	177.2	15.8	45.2
900 [482]	60	206.1	190.4	11.4	40.6
900 [482]	60	200.5	189.3	13.4	40.0
900 [482]	120	212.2	192.9	16.3	35.7
950 [510]	30	179.4	164.5	17.2	50.5
950 [510]	45	190.3	172.2	16.9	45.7
950 [510]	60	195.2	174.8	15.8	40.6
950 [510]	60	197.5	186.4	13.7	37.8
950 [510]	60	195.2	183.5	13.5	37.6
950 [510]	156	207.6	187.0	14.8	37.0
1000 [538]	45	187.8	167.7	18.2	45.6
1000 [538]	60	188.4	175.8	15.8	44.0
1000 [538]	60	188.3	175.7	16.8	44.6

Table 6: Tensile Testing Results for Embodiments of the Present Invention with 15% Cold Work Reduction

Table 7 lists embodiments of the present invention wherein test pieces were hot rolled, cold drawn to 19%, and direct aged. Additionally, the embodiments described in Table 7 were annealed and centerless ground prior to cold drawing.

Aging Temp. (°F) [°C]	Aging Time (minutes)	UTS (ksi)	UTS (MPa)	0.2% YS (ksi)	0.2% YS (MPa)	Elong. (%)	RA (%)	Modulus
as-drawn	0	153.3	1057	141.0	972	13	57	13.3
950 [510]	166	210.2	1449	193.3	1333	12	27	14.2
950 [510]	170	209.4	1444	191.6	1321	14	31	14.7
1000 [538]	72	191.7	1322	173.8	1198	22	47	15.4
1000 [538]	89	196.9	1357	179.3	1236	19	32	15.3
1000 [538]	125	196.5	1355	179.1	1235	14	33	14.1
1000 [538]	200	196.0	1351	178.6	1231	15	40	14.4
1050 [565]	81	183.8	1267	166.6	1149	22	54	14.1
1050 [565]	88	186.3	1284	169.0	1165	23	52	15.1
1050 [565]	133	183.1	1262	165.4	1140	20	54	13.6
1050 [565]	182	181.7	1253	164.5	1134	20	50	15.1

Table 7: Tensile Testing Results for Embodiments of the Present Invention with 19% Cold Work Reduction

Table 8 lists embodiments of the present invention wherein test pieces were hot rolled, cold drawn to 20% reduction, and direct aged.

- 5 Additionally, the test pieces of Table 8 were not annealed and centerless ground prior to cold drawing. The embodiments of the present invention in Table 8 produced an increase in UTS of approximately 5% and an increase in 0.2% yield strength of 6% over the embodiments employing a cold work of 15% reduction. Cold working to 20% reduction reduced ductility by either 5% (as measured by elongation) or 9% (as measured by RA).
- 10

Aging Temp (°F) [°C]	Aging Time (minutes)	UTS (ksi)	0.2% YS (ksi)	Elong. (%)	RA (%)
-	0	155.2	152.0	16.4	63.5
900 [482]	45	201.1	185.9	15.3	40.6
900 [482]	120	216.0	199.4	9.3	36.4
950 [510]	30	188.1	173.9	17.3	50.3
950 [510]	45	200.8	184.0	17.4	43.8
950 [510]	60	205.0	187.2	13.2	36.9
950 [510]	156	214.8	196.3	14.5	32.5
1000 [538]	45	194.2	174.7	17.2	40.4
1000 [538]	60	196.5	176.9	18.0	40.0

Table 8: Tensile Testing Results for Embodiments of the Present Invention with 20% Cold Work Reduction

- Table 9 lists embodiments of the present invention wherein test pieces were hot rolled, cold drawn to 25% reduction, and direct aged.
- 15 Additionally, the embodiments described in Table 9 were not annealed and

centerless ground prior to cold drawing. The embodiments of the present invention listed in Table 9 on average show an increase in UTS of approximately 7% and an increase in 0.2% yield strength of 9% over the embodiments which utilized cold working to 15% reduction. Cold working to 25% reduction reduced ductility by either 11% (as measured by elongation) or 2% (measured by RA) as compared to embodiments utilizing cold working to 15% reduction.

Aging Temp (°F) [°C]	Aging Time (minutes)	UTS (ksi)	0.2% YS (ksi)	Elong. (%)	RA (%)
-	0	162.5	159.4	16.9	64.0
900 [482]	45	207.2	193.0	13.7	43.8
900 [482]	120	220.9	204.6	15.2	34.9
950 [510]	30	194.2	180.8	16.9	48.7
950 [510]	45	205.1	189.9	15.4	43.2
950 [510]	60	207.6	189.3	14.0	39.4
950 [510]	156	212.7	193.7	16.4	33.8
1000 [538]	45	199.3	181.7	16.0	46.5

Table 9: Tensile Testing Results for Embodiments of the Present Invention with 25% Cold Work Reduction

The tensile properties of embodiments of the present invention including the step of cold working to 13% or 15% reduction are shown graphically in Figures 1 to 3. Figure 1 graphically depicts the effect of aging time on samples of Ti-38-644 beta titanium alloy having the composition shown in Table 1 and wherein the method included a step of cold working to a 13 or 15% reduction. The UTS and 0.2% yield strength increase rapidly for at least the first 60 minutes of total aging time. For these embodiments, UTS of the test pieces reached 180 ksi in approximately 30 minutes of total aging time. These test pieces were aged in a conventional laboratory testing oven. Production aging furnaces would likely more efficiently heat articles and, therefore, in a production furnace it is expected that total aging times in the method of the present invention necessary to reach high strength (180 ksi, for example) may be reduced, possibly by two thirds or more in some cases.

The aging of the beta titanium alloy may be conducted at an temperature below the beta transus. Preferably, the aging of the beta titanium alloy occurs at a temperature between 800°F (427°C) and 1100°F (538°C). For

some applications, the aging of the beta titanium alloy may occur between 800°F (427°C) and 1000°F (538°C), and more preferably between 900°F (482°C) and 1000°F (538°C).

It can be seen in Figure 1 that ductility of the test pieces, as measured by elongation or RA, decreased with total aging time. However, ductility decreases slowly with total aging time, and UTS of over 200 ksi was achieved while maintaining relatively good ductility. For certain uses, such as in the production of suspension springs for automobile, snow mobile, motorcycle and other recreational vehicles and valve springs for piston engines, short aging times are preferred. Automobile production lines may include installations for winding and aging springs as required for production. The springs may be, for example, wound and then aged on a conveyer belt as the belt passes through an aging furnace. Preferably in these and other applications, aging of the beta-titanium alloy will be for a period of less than 3 hours. More preferably the aging of the beta-titanium alloy will be for a period of less than 2 hours, and even more preferably for some time sensitive applications the aging will be for a period of less than 1 hour or more preferably for less than 45 minutes. Alloys produced by the present invention may also be useful in other applications than springs, such as, for example, in the biomedical industry for surgical instruments or implants.

Figure 2 depicts the effect of aging time and temperature on UTS of test pieces of the beta titanium alloy of Table 1 processed by embodiments of the present invention including cold working to 13% or 15% reduction. The embodiments of the present invention employing aging at lower temperatures achieved higher UTS. This may be expected due to crystalline growth at higher temperatures and the lower volume of alpha phase present in the alloy as a result of the processing conditions, which both may adversely effect the strength of a beta titanium alloy.

Figure 3 depicts the effect of aging time and temperature on ductility of test pieces of the beta titanium alloy of Table 1, as measured by reduction in area, using embodiments of the present invention including cold working to 13% or 15% reduction. The embodiments of the present invention utilizing aging at higher temperatures produced higher ductility in the test pieces over time. This

may be expected due to crystalline growth at higher temperatures which, although adversely effecting strength, enhances ductility of the beta titanium alloy.

A second titanium ingot was produced and processed according to method of the present invention. The composition of the second ingot at three locations is shown in Table 10. The composition of the alloy was tested in three locations to verify the composition and ensure a fairly consistent composition throughout the ingot.

Source	Ti	Al	V	Cr	Zr	Mo	O	Fe	C	N
Top of Second Ingot	Bal.	3.65	7.95	6.16	4.06	4.08	0.1	0.05	0.01	0.01
Middle of Second Ingot	Bal.	3.45	7.9	6.29	4.12	4.04	0.1	0.06	0.02	0.01
Bottom of Second Ingot	Bal.	3.34	7.85	6.43	4.14	3.98	0.1	0.06	0.01	0.01

Table 10: Composition of Second Ingot

The second ingot was processed according to the method of the present invention. The second ingot was hot rolled at a temperature not to exceed 1825°F (996°C), annealed and air cooled. With regard to Table 11, test pieces produced from the second ingot were hot rolled, cold drawn to provide 16.5% reduction and then direct aged at the temperatures and for the times shown in the table. The test pieces described in Table 11 also were annealed at a temperature not to exceed 1450°F (774°C) and air cooled prior to cold drawing. The embodiments listed in Table 11 produced higher strength (UTS greater than 190 ksi) and maintained ductility (greater than 8% elongation and greater than 20% RA) with less than 30 minutes of direct aging. UTS values greater than 200 ksi and as high as 220 ksi were realized in the listed embodiments. Again, the highest UTS values were realized at the lower aging temperatures, 900°F (482°C), at which a UTS of 220 ksi was achieved at a total aging time of only 60 minutes. The highest ductility as measured by elongation and RA was realized at the higher aging temperature of 1050°F (566°C).

Aging Temp (°F) [°C]	Aging Time (minutes)	UTS (ksi)	UTS (MPa)	0.2% YS (ksi)	0.2% YS (MPa)	Elong. (%)	RA (%)
NA	0	164.2	1132	150.8	1040	16.1	52.5
NA	0	154.6	1066	149.2	1029	17.9	52.9
900 [482]	30	205.5	1417	191.0	1317	11.5	33.3
900 [482]	45	207.6	1431	191.7	1322	11.5	31.4
900 [482]	45	216.0	1489	197.7	1363	10.9	29.1
900 [482]	60	220.4	1519	202.7	1397	11.0	30.4
900 [482]	60	216.2	1490	201.1	1386	10.5	28.0
NA	0	164.2	1132	150.8	1040	16.1	52.5
NA	0	154.6	1066	149.2	1029	17.9	52.9
950 [510]	30	198.7	1370	182.7	1260	13.7	35.9
950 [510]	30	198.7	1370	181.3	1250	14.3	35.0
950 [510]	45	207.0	1427	191.7	1322	13.7	32.0
950 [510]	45	205.1	1414	190.5	1313	12.6	30.8
950 [510]	60	210.5	1451	192.6	1328	13.8	24.7
950 [510]	60	209.3	1443	193.5	1334	13.1	29.8
NA	0	164.2	1132	150.8	1040	16.1	52.5
NA	0	154.6	1066	149.2	1029	17.9	52.9
1000 [538]	30	190.9	1316	175.2	1208	17.6	37.0
1000 [538]	45	197.8	1364	182.4	1257	14.0	36.8
1000 [538]	45	199.9	1378	182.9	1261	20.4	35.1
1000 [538]	60	201.5	1389	185.1	1276	-	34.5
1000 [538]	60	204.7	1411	189.5	1306	16.0	39.5

Table 11: Tensile Testing Results for Embodiments of the Present Invention produced from the Second Ingot with 16.5% Cold Work Reduction

Generally, the test pieces produced by an embodiment of the process of the present invention as described in Table 11 achieved higher tensile strengths with shorter aging times than the test pieces produced by the embodiments of the process of the present invention as described in Tables 3 to 9. However, generally, the ductility of the test pieces described in Table 11 were lower. It is believed that the higher hot rolling temperature experienced by the second ingot produced the lower ductility since the higher processing temperatures favored a larger prior beta grain size. The higher strength is thought to be associated with slower cooling after the anneal which allowed for some aging prior to cold working.

Table 12 shows the results of Rotating Beam Fatigue Testing on articles prepared by the method of the present invention wherein the articles were hot rolled, cold drawn to 15% reduction, and direct aged at 950°F (510°C) for one hour. The Rotating Beam Fatigue Testing was conducted to determine the

bending fatigue according to international testing standard ISO 1143 at a frequency of 50 Hz, R= -1 and using a smooth bar. The results indicate the number of cycles experienced for each specimen prior to failure or the total number of cycles performed on the specimen if no failure occurred.

5

Max Stress ksi	Max Stress Mpa	Cycles	Comment
73	500	13401000	Run out, no failure
83	575	10017100	Run out, no failure
87	600	10804700	Run out, no failure
87	600	151900	Failure
91	625	620800	Grip Fail
94	650	525100	Failure
98	675	79300	Failure
102	700	395200	Failure

Table 12: Rotating Beam Fatigue Testing Results for Embodiments of the Present Invention comprising 15% Cold Work Reduction and direct aging at 950°F (510°C) for one hour.

Table 13 shows the results of Load Controlled Axial Fatigue Testing on articles prepared by the method of the present invention wherein the articles were hot rolled, cold drawn to 15% reduction, and direct aged at 950°F (510°C) for one hour. The Load Controlled Axial Fatigue Testing was conducted to determine the fatigue of the articles according to ASTM E-466-96 with a frequency of 29 Hz at R= 0.1. The results indicate the number of cycles experienced for each specimen prior to failure. Specimen prepared using different conditions in the method of the present invention, such as, a longer aging time, different aging temperature or different degree of cold working, for example, may result in an increase in the number of cycles prior to failure in the fatigue tests.

Max Stress ksi	Max Stress MPa	Cycles	Comment
142	979	2313507	Failure
145	1000	286613	Failure
150	1034	170773	Failure
160	1103	22532	Failure

Table 13: Load Controlled Axial Fatigue Testing Results for Embodiments of the Present Invention comprising 15% Cold Work Reduction and direct aging at 950°F (510°C) for one hour.

- 5 Though the method of the present invention is described above with respect to beta titanium alloys of certain compositions, it is believed that the method of the present invention has wider application, to the processing of other beta titanium alloys. For example, without limiting the method of the present invention, some additional commercially available beta titanium alloys that may
- 10 benefit from the present invention are titanium alloys having the following nominal compositions, in weight percentages. Ti-12Mo-6Zr-2Fe (an alloy comprising 12% molybdenum, 6% zirconium, 2% iron and titanium, and which is available commercially in at least one form as ALLVAC TMZF alloy); Ti-4.5Fe-6.8Mo-1.5Al (an alloy comprising 4.5% iron, 6.8% molybdenum, 1.5% aluminum and titanium,
- 15 and which is available commercially in at least one form as TIMETAL LCB alloy); Ti-15Mo-2.6Nb-3Al-0.2Si (an alloy comprising 15% molybdenum, 2.6% niobium, 3% aluminum, 0.2% silicon and titanium, and which is available commercially in at least one form as TIMETAL 21S alloy); Ti-15V-3Cr-3Sn-3Al (an alloy comprising 15% vanadium, 3% chromium, 3% tin, 3% aluminum and titanium, and which is
- 20 available commercially in at least one form as ALLVAC 15-3 alloy), Ti-11.5Mo-6Zr-4.5Sn (an alloy comprising 11.5% molybdenum, 6% zirconium, 4.5 tin and titanium, and which is available commercially in at least one form as UNITEK Beta III alloy); and Ti-6V-6Mo-5.7Fe-2.7Al (an alloy comprising 6% vanadium, 6% molybdenum, 5.7% iron, 2.7 aluminum and titanium, and which is available
- 25 commercially in at least one form as TIMETAL 125 alloy). The compositions of the alloys presented above are nominal compositions, and the content of each element may vary by at least as much as 2% or more and the alloys may also include additional components.

It is to be understood that the present description illustrates those aspects of the invention relevant to a clear understanding of the invention. Certain aspects of the invention that would be apparent to those of ordinary skill in the art and that, therefore, would not facilitate a better understanding of the invention have not been presented in order to simplify the present description. Although the present invention has been described in connection with certain embodiments, those of ordinary skill in the art will, upon considering the foregoing description, recognize that many modifications and variations of the invention may be employed. All such variations and modifications of the invention are intended to be covered by the foregoing description and the following claims.

1. A method for processing titanium alloys, the method comprising:
cold working a beta titanium alloy; and
direct aging the beta titanium alloy for a total aging time of less than
5 4 hours.
2. The process of claim 1, wherein the beta titanium alloy comprises at least one of aluminum, vanadium, molybdenum, chromium, and zirconium.
3. The process of claim 1, further comprising:
hot rolling the beta titanium alloy prior to cold working the beta
10 titanium alloy.
4. The process of claim 3, wherein cold working the beta titanium alloy comprises cold working the beta titanium alloy to at least a 5% reduction.
5. The process of claim 4, wherein cold working the beta titanium alloy comprises cold working the beta titanium alloy to at least a 15% reduction.
- 15 6. The process of claim 5, wherein cold working the beta titanium alloy comprises cold working the beta titanium alloy to less than 60% reduction.
7. The process of claim 6, wherein cold working the beta titanium alloy comprises cold working the beta titanium alloy to less than 35% reduction.
8. The process of claim 7, wherein cold working the beta titanium alloy
20 comprises cold working the beta titanium alloy to less than 20% reduction.
9. The process of claims 1, wherein direct aging the beta titanium alloy comprises direct aging the beta titanium alloy in the temperature range about 800°F (427°C) to about 1200°F (649°C).
10. The process of claim 1, wherein direct aging the beta titanium alloy
25 comprises direct aging the beta titanium alloy in the temperature range of about 800°F (427°C) to about 1000°F (538°C).
11. The process of claim 1, wherein direct aging the beta titanium alloy comprises direct aging the beta titanium alloy in the temperature range about 900°F (882°C) to about 1000°F (538°C).
- 30 12. The process of claim 1, wherein direct aging the beta titanium alloy comprises direct aging the beta titanium alloy for less than 3 hours.

13. The process of claim 1, wherein the direct aging the beta titanium alloy comprises direct aging the beta titanium alloy for less than 2 hours.
14. The process of claim 1, wherein direct aging the beta titanium alloy comprises direct aging the beta titanium alloy for less than 1 hour.
- 5 15. The process of claim 1, wherein the direct aging the beta titanium alloy comprises direct aging the beta titanium alloy for less than 45 minutes.
16. The process of claim 1, wherein the beta titanium alloy comprises, by weight, 3.0 to 4.0% aluminum, 7.5 to 8.5 % vanadium, 5.5 to 6.5 % chromium, 3.5 to 4.5 % molybdenum, 3.5 to 4.5% zirconium 3.5 to 4.5 %, and titanium.
- 10 17. A process for producing an article of manufacture, comprising:
providing a beta titanium alloy comprising, by weight, 3.0 to 4.0% aluminum, 7.5 to 8.5% vanadium, 5.5 to 6.5 % chromium, 3.5 to 4.5 % molybdenum, 3.5 to 4.5% zirconium, and titanium;
hot working the beta titanium alloy;
15 cold working the beta titanium alloy to provide a 5 to 60% reduction;
direct aging the beta titanium alloy for a total aging time of less than 2 hours at a temperature in the range about 800°F (427°C) to about 1100°F(593°C).
18. The process of claim 17, wherein the article of manufacture is a spring.
- 20 19. The process of claim 17, wherein cold working the beta titanium alloy comprises drawing the beta titanium alloy through a die.
20. The process of claim 17, wherein hot working the beta titanium alloy comprises forming the beta titanium alloy into a bar, a rod, or a coil.
21. The process of claim 17, wherein direct aging the beta titanium alloy
25 includes a total aging time less than one hour.
22. The process of claim 17, wherein direct aging the beta titanium alloy includes a total aging time less than forty-five minutes.
23. The process of claim 22, wherein direct aging the beta titanium alloy includes a direct aging temperature in the range about 900°F (482°C) to about
30 1000°F (538°C).
24. The process of claim 18, wherein the spring is a component of an automobile, snowmobile, motorcycle, recreational vehicle, or engine.

25. The process of claim 17, further comprising:

centerless grinding the beta titanium alloy; and

annealing the beta titanium alloy prior to cold working the beta titanium alloy.

5 26. The process of claim 25, wherein cold working the beta titanium alloy comprises drawing the beta titanium alloy through a die.

27. The process of claim 17, wherein the cold working of the alloy provides a 5 to 35% reduction.

28. A method of processing a titanium alloy, comprising:

10 direct aging a cold worked beta titanium alloy for a total aging time of less than 4 hours.

29. The process of claim 28, wherein the beta titanium alloy comprises at least one of aluminum, vanadium, molybdenum, chromium, and zirconium.

30. The process of claim 28, further comprising:

15 hot rolling the beta titanium alloy prior to cold working the beta titanium alloy.

31. The process of claims 28, wherein direct aging the beta titanium alloy comprises direct aging the beta titanium alloy in the temperature range about 800°F (427°C) to about 1200°F (649°C).

20 32. The process of claim 28, wherein direct aging the beta titanium alloy comprises direct aging the beta titanium alloy in the temperature range of about 800°F (427°C) to about 1000°F (538°C).

33. The process of claim 28, wherein direct aging the beta titanium alloy comprises direct aging the beta titanium alloy in the temperature range about 25 900°F (882°C) to about 1000°F (538°C).

34. The process of claim 28, wherein direct aging the beta titanium alloy comprises direct aging the beta titanium alloy for less than 3 hours.

35. The process of claim 28, wherein the direct aging the beta titanium alloy comprises direct aging the beta titanium alloy for less than 2 hours.

30 36. The process of claim 28, wherein direct aging the beta titanium alloy comprises direct aging the beta titanium alloy for less than 1 hour.

37. The process of claim 28, wherein the direct aging the beta titanium alloy comprises direct aging the beta titanium alloy for less than 45 minutes.
38. The process of claim 28, wherein the beta titanium alloy comprises, by weight, 3.0 to 4.0% aluminum, 7.5 to 8.5 % vanadium, 5.5 to 6.5 % chromium, 3.5 to 4.5 % molybdenum, 3.5 to 4.5% zirconium 3.5 to 4.5 %, and titanium.
39. An article of manufacture prepared by a process, comprising:
cold working an article, wherein the article comprises a beta titanium alloy; and
direct aging the article for a total aging time of less than 4 hours.
40. The article of manufacture of claim 39, wherein the article is one of a bar, a rod, or a coil.
41. The article of manufacture of claim 39, wherein the beta titanium alloy comprises, by weight, 3.0 to 4.0% aluminum, 7.5 to 8.5 % vanadium, 5.5 to 6.5 % chromium, 3.5 to 4.5 % molybdenum, 3.5 to 4.5% zirconium 3.5 to 4.5 %, and titanium.
42. The article of manufacture of claim 39, wherein direct aging the beta titanium alloy comprises direct aging the beta titanium alloy in the temperature range about 800°F (427°C) to about 1200°F (649°C).
43. The article of manufacture of claim 39, wherein the direct aging the beta titanium alloy comprises direct aging the beta titanium alloy for less than 2 hours.
44. The article of manufacture of claim 39, wherein direct aging the beta titanium alloy comprises direct aging the beta titanium alloy for less than 1 hour.
45. The article of manufacture of claim 39, wherein the direct aging the beta titanium alloy comprises direct aging the beta titanium alloy for less than 45 minutes.

FIGURE 1

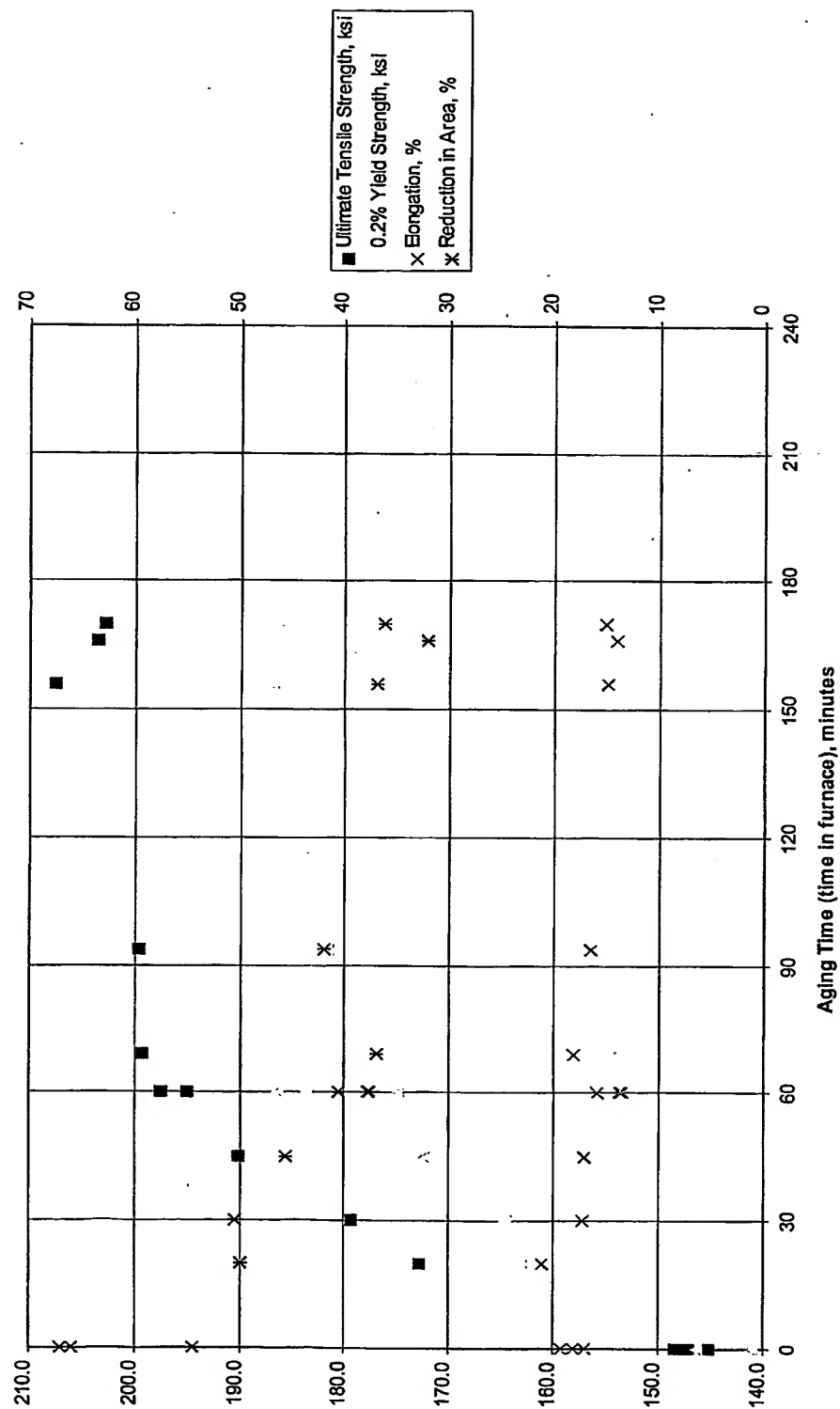


FIGURE 2

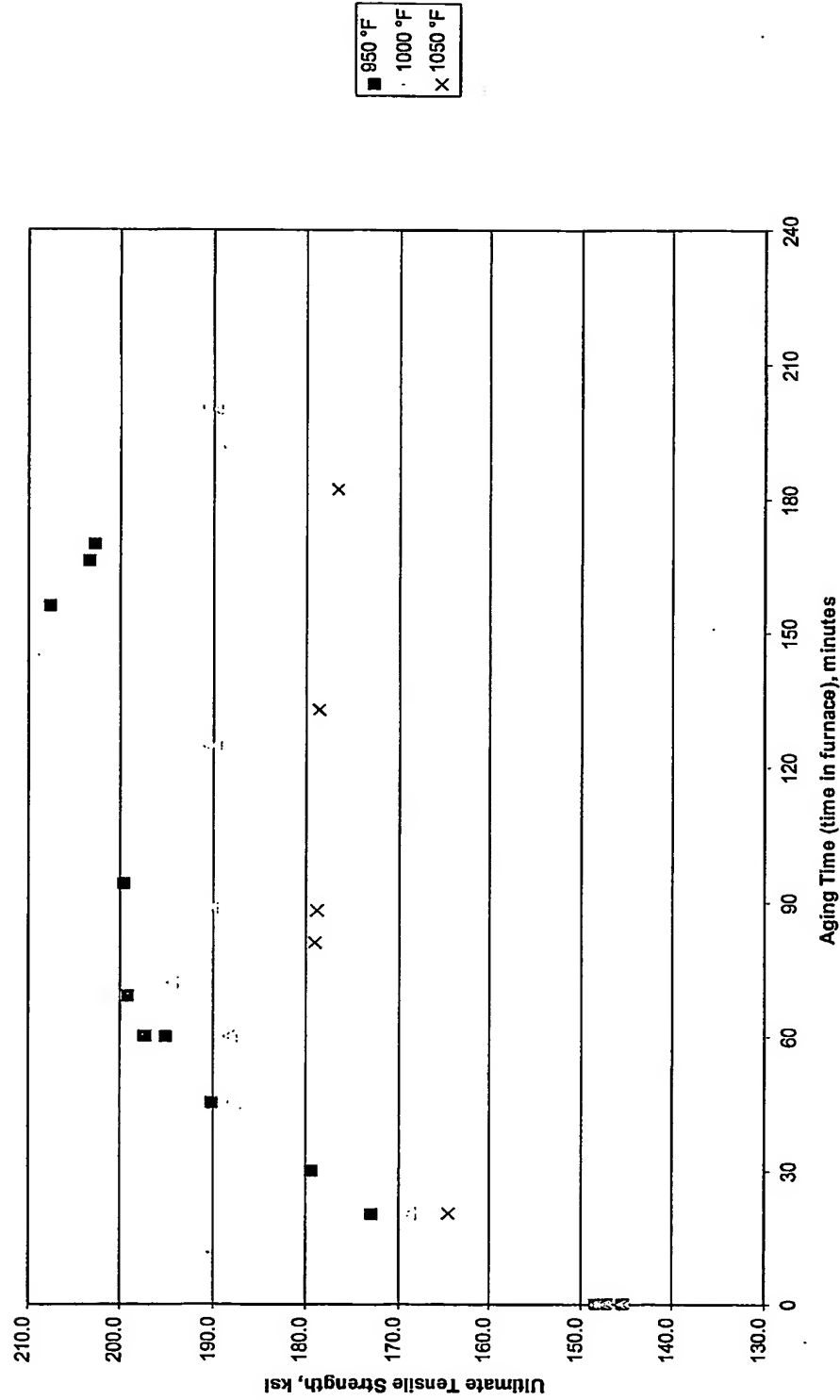
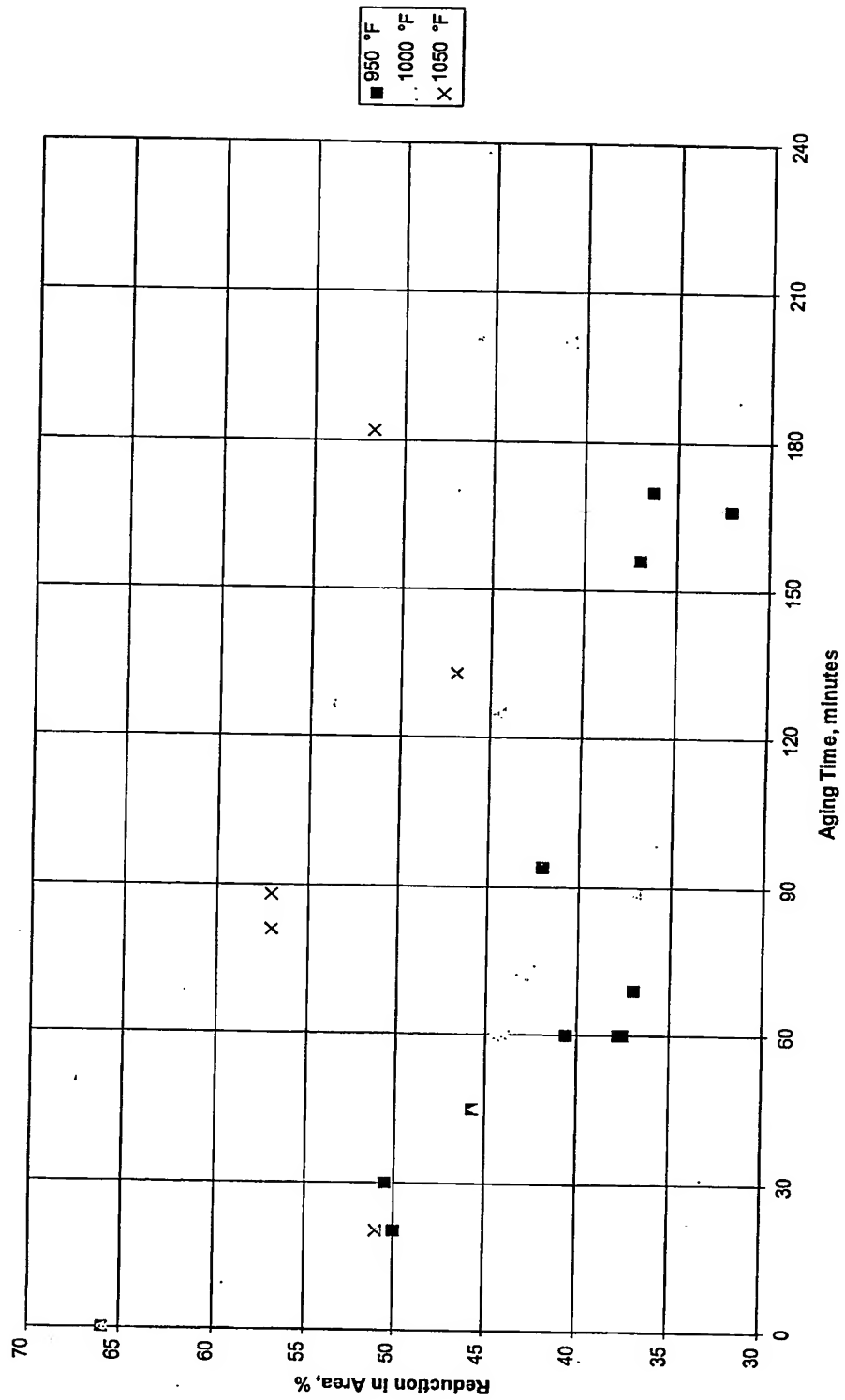


FIGURE 3



INTERNATIONAL SEARCH REPORT

International application No.

PCT/US02/18269

A. CLASSIFICATION OF SUBJECT MATTER

IPC(7) : C22F 1/18

US CL : 148/671

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 148/671, 670, 421

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
Please See Continuation Sheet

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 6,258,182 B1 (SCHETKY et al) 10 July 2001 (10.07.2001), abstract, Figure 7, column 3, lines 13-14 and 42-50, column 8, lines 24-25, 44-45 and 54-57, column 9, lines 3-10.	1-2, 4-8, 12-13, 28-29, 34-35 and 39-45

Y		
Y	JP 01-279736 A (NIPPON MINING CO. LTD.) 10 November 1989 (10.11.1989), abstract.	3, 14-16, 30 and 36-38
Y	US 6,250,812 B1 (UEDA et al) 26 June 2001 (26.06.2001), abstract, Figure 1, 6, 7 and 8, column 4, lines 20-51, column 14, lines 31-45, Table 9, columns 31-34.	1-13, 16, 28-35 and 38-45
Y	US 5,358,586 A (SCHUTZ et al) 25 October 1994 (25.10.1994), abstract, Figure 1, column 1, lines 15-44.	1-16 and 28-45
		3, 16, 30 and 38

☐ Further documents are listed in the continuation of Box C.

☐ See patent family annex.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier application or patent published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T"

later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X"

document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y"

document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&"

document member of the same patent family

Date of the actual completion of the international search

18 October 2002 (18.10.2002)

Date of mailing of the international search report

02 JAN 2003

Name and mailing address of the ISA/US
Commissioner of Patents and Trademarks
Box PCT
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INTERNATIONAL SEARCH REPORT

International application No.
PCT/US02/18269

Box I Observations where certain claims were found unsearchable (Continuation of Item 1 of first sheet)

This international report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claim Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:
2. ☐ Claim Nos.:
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:
3. ☐ Claim Nos.:
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box II Observations where unity of invention is lacking (Continuation of Item 2 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.
2. ☐ As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.
3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:
4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

☐
☐

- The additional search fees were accompanied by the applicant's protest.
No protest accompanied the payment of additional search fees.

INTERNATIONAL SEARCH REPORT

PCT/US02/18269

Continuation of B. FIELDS SEARCHED Item 3:

EAST

search terms: beta titanium, work\$, deform\$, Ti-38-644, AMS 4957A, AMS 4958A, ag\$4